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(54) PROCESS FOR PRODUCING AMINOPLAST RESIN-IMPREGNATED SUPPORT WEBS SUITABLE FOR DECORATIVE SURFACE IMPROVEMENT OF WOODEN BOARDS

(71) We, TH. GOLDSCHMIDT A.G. a German Body Corporate of 43 Essen, Goldschmidtstrasse 100, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for producing aminoplast resin-impregnated support webs or substrates suitable for decorative surface improvement of wooden boards or sheets.

It is already known e.g. from Belgian Patent Specification No. 729,117 to impregnate and coat support webs, particularly those of paper, with solutions of aminoplast resins. The aminoplast resins used in these processes may be melamine/formaldehyde precondensate resins or urea/formaldehyde precondensate resins or mixtures thereof.

It is also known to utilise resins of differing flowability or differing curing characteristics for the impregnation and coating. Thus, German Patent Specification No. 1,053,303 describes a process wherein the support web is first impregnated (soaked) with the solution of a resin which is highly flowable under pressure and then, after optional drying, coated with the solution of a resin which is less flowable under pressure and which hardens rapidly.

Upon comparing the surfaces obtained by curing condensation resins based on aminoplast resins it is found that the surfaces obtained by curing melamine/formaldehyde condensation resins are of better quality than surfaces obtained by curing urea/formaldehyde condensation resins. Consequently, melamine/formaldehyde condensation resins are generally used for surface improvement purposes.

However, compared to urea, melamine has the disadvantage of being more expensive. This invention is concerned with partly replacing melamine/formaldehyde condensation resins by urea/formaldehyde condensation resins to reduce cost without having to accept the known disadvantages resulting from the use of urea resins such as, for example, less satisfactory resistance to chemicals and atmospheric influences as well as difficulties in the formation of the surface.

The present invention provides a process for the production of an aminoplast resin impregnated support web of cellulosic material, suitable for decorative surface improvement of sheets of wooden material, which comprises impregnating the web with a solution of a mixture of urea/formaldehyde and melamine/formaldehyde condensation resins, the degree of condensation of the urea/formaldehyde condensation resin being such that a 50% by weight solution of the resin in water has a viscosity not greater than 70 cP at 20°C and the degree of condensation of the melamine/formaldehyde resin being such that a 50% by weight solution of the resin in water has a viscosity of at least 100 cP at 20°C, and coating the web with a melamine/formaldehyde condensation resin.

It is preferred to use a resin mixture for impregnation which consists of 50 to 90 weight percent urea/formaldehyde condensation resin, the balance being melamine/formaldehyde condensation resin.

It is believed that the urea resin preferentially penetrates into the cellulosic support web and that the more condensed melamine resin concentrates more at the surface of the support web where it is finally covered by the melamine/formaldehyde coating resin. It is particularly surprising that it is possible to

utilise in the impregnation resin as much as 50% and even up to 90% by weight urea resin and only 10% by weight of melamine resin. With such a concentration ratio, it was to be expected that, in view of the great mutual affinity of the two resins, the urea resin would migrate into the upper coating layer so causing the above described difficulties.

A urea to formaldehyde molar ratio of 1:1.5 to 1:2.5 is preferably used for producing the urea/formaldehyde condensate while a molar ratio of 1:1.5 to 1:3.5 is preferably used to prepare the melamine/formaldehyde condensate.

It may be of special advantage to keep the concentration of the resin mixture in the solution of impregnating resin at a low level so as to attain a particularly intimate bonding and anchoring of the impregnating resin with the cellulosic support web. This is believed to result in the urea resin preferentially penetrating into the cellulose fibre and becoming particularly well fixed in the subsequent drying process.

It is possible to produce the area resin and the melamine resin by methods known *per se* sequentially in one and the same reaction vessel.

The relative difference in condensation degree can be secured by first reacting urea with the requisite amount of formaldehyde in an alkaline medium, this mixture is then subjected to a first condensation under acidic conditions until the desired condensation degree has been obtained, and further condensation is prevented by making this reaction solution alkaline again. The melamine/formaldehyde condensate can then be produced in alkaline medium. By this method, the degree of condensation of the urea/formaldehyde condensation resin first obtained remains substantially constant and the degree of condensation of the melamine/formaldehyde condensation resin can be adjusted as desired. The reaction periods depend on the values previously determined for the individual resins.

When the surfaces of sheets of wooden material are decorated with the resin impregnated and coated support webs obtained in accordance with the invention, surfaces are obtained which are equivalent to pure melamine resin films as regards their hydrolysis resistance, despite the urea content in the impregnating resin. This represents an appreciable reduction in the cost of the product of the process. The products of the process can be used in the up-to-date so-called brief-time presses, i.e. at relatively short pressing times, elevated temperature and a large number of pressing cycles per unit of time.

A further aspect of the invention provides a method of decorative surface finishing of sheets of wooden material which comprises laminating a web according to the invention onto the surface of the sheet.

The following Examples are given to illustrate the invention. The preparation of the resins is described in the preliminary Experiments.

Experiment A

Production of urea formaldehyde condensate.

A reaction vessel equipped with thermometer, stirrer, reflux condenser and a device for continuously measuring the pH value is used. 800 g of 30% formaldehyde are introduced and adjusted to pH 9 with 3 M sodium hydroxide. 209 g of urea are then added and the reaction mixture is heated to 90°C. During a reaction period of 10 minutes, the pH value of 9 is maintained constant by repeated additions of 3 M sodium hydroxide. The mixture is then cooled to 80°C, the pH value is adjusted to 4 with 3 M phosphoric acid, the mixture is heated to 90°C and condensed at this temperature for 1/4 hour. The mixture is now mixed with a further 31 g urea, the pH is again adjusted to 9 with 3 M sodium hydroxide and while maintaining this pH constant the formulation is condensed for 1/4 hour. After cooling, a urea/formaldehyde resin having a pH value of 8.3 (20°C) is obtained. The viscosity of a 50% by weight solution of the resin in water at 20°C is 60 cp.

Experiment B

Production of a melamine formaldehyde condensate.

In a reaction vessel equipped as described in Experiment A, 660 g of 30% formaldehyde are adjusted to pH 9 with 1 M sodium hydroxide, 378 g melamine are added and the reaction mixture is heated to 94° with thorough stirring. After a reaction period of 2 hours, at 94°C the mixture is cooled. A melamine/formaldehyde condensate having a pH value of 9.2 at 20°C, is obtained. The viscosity of a 50% by weight solution of the resin in water at 20°C is 125 cP.

Experiment C

Preparation of a urea/melamine formaldehyde condensate

In a reaction vessel similar to that described in Experiment A, 1200 g of 20% formaldehyde are adjusted to pH 9 with 1 M sodium hydroxide, 180 g urea are added

and the reaction mixture is heated to 90°C. The pH value of 9 is maintained constant by repeated additions of 1 M sodium hydroxide during a reaction period of 10 minutes. The mixture is then cooled to 80°C, the pH is adjusted to 4 with 3 M phosphoric acid, the mixture is heated to 90°C and condensed for 1/4 hour at this temperature. After cooling to 70°C the pH is adjusted to 9 with 1 M sodium hydroxide, 63 g melamine are added and, when the melamine has dissolved, the mixture is heated to 94°C. Condensation of the melamine into the present reaction mixture is continued at this temperature until the condensate mixture obtained has a pH value of 8.5. The viscosity of a 50% by weight solution of the resin in water at 20°C is 90 cP.

In the following Examples impregnation resins having the following compositions are used:

Impregnating resin I

60 parts of the urea resin prepared in Experiment A

40 parts of melamine resin prepared in Experiment B

Impregnating resin II

90 parts of urea resin prepared in Experiment A

10 parts of melamine resin prepared in Experiment B

Impregnating resin III

100 parts of the resin mixture prepared in Experiment C.

EXAMPLE 1

200 parts by weight of impregnating resin I are thoroughly mixed with 3 parts by weight of a 50% N-methylethanolammoniumacetate solution (latent curing agent) and 1 part by weight of a separation medium based on mineral oil (100% content of active material). A white, pigmented, absorptive, fine celloluse paper having a surface weight of 80 g/m² is saturated with this resin mixture and heat-treated in a drying apparatus until the impregnated paper web has a content of 12% w/w volatile constituents. The content of volatile constituents is the loss in weight which the impregnated paper web suffers in a heat treatment at 160°C for 10 minutes. The resin-impregnated paper now has a surface weight of 130 g/m². This resin-impregnated support web is then coated uniformly on both sides with a melamine/formaldehyde resin solution. This melamine resin solution has a solids content of 55% w/w, a molar ratio melamine to formaldehyde of 1:2, a viscosity of 100 cP, a precipitation number of 1.0 and is mixed with the same additives as impregnating resin I. The impregnated and coated web is again adjusted to a volatile constituents content of 7% w/w by drying. The surface weight of the resulting impregnated and coated, white, decorative film is 200 g/m².

EXAMPLE 2

200 parts by weight of the impregnating resin II are mixed with the same additives as in Example 1 and, after intensive mixing, are used for impregnating a white, pigmented, absorptive, fine cellulose paper having a surface weight of 120 g/m². By following the procedure of Example 1, there is obtained an impregnated support web having a surface weight of 185 g/m² at a content of volatile constituents of about 12% w/w. By coating the impregnated paper with the melamine resin as described in Example 1, an impregnated and coated support web having a surface weight of 270 g/m² is obtained.

EXAMPLE 3

200 g of the impregnating resin III are reacted with 2.5 parts by weight of a 50% N-methylethanolammonium acetate solution (latent curing medium) and 1 part by weight of a mineral oil based separation medium (100% content of active material) and thoroughly mixed.

By following the procedure described in Example 1, an impregnated and coated support web having a surface weight of 200 g/m² and a volatile constituents content of 6.5% w/w is produced.

EXAMPLE 4

The impregnated and coated webs produced in Examples 1, 2 and 3 are laminated onto the surface of a 16 mm thick chipboard. The conditions of pressing are 8 minutes at 145°C at the heating platen of the press using 20 kp/cm² pressure, an asbestos cushion and back cooling. The pressing is against chromium plated brass plates of high mirror finish.

The resulting laminates have uniformly mirror-finished surface coatings whose physical and chemical properties are indistinguishable from those of surface coatings produced by using decorative films based on pure melamine resins.

EXAMPLE 5

The decorative films produced in Examples 1, 2 and 3 are laminated onto the surface of a 16 mm thick chipboard. The pressing conditions are 160°C at the heating platen for 150 seconds, using 18 kp/cm² pressure, an asbestos cushion but no back cooling. Pressing is against chromium

plated brass sheets of reduced degree of polish.

The resulting surface coatings have an optically uniform appearance and, upon comparison with surface coatings produced using decorative films based on pure melamine resin, cannot be distinguished by their physical or chemical properties.

WHAT WE CLAIM IS:—

1. Process for the production of an aminoplast resin impregnated support web of cellulosic material, suitable for decorative surface improvement of sheets of wooden material, which comprises impregnating the web with a solution of a mixture of urea/formaldehyde and melamine/formaldehyde condensation resins, the degree of condensation of the urea/formaldehyde condensation resin being such that a 50% by weight solution of the resin in water has a viscosity not greater than 70 cP at 20°C and the degree of condensation of the melamine/formaldehyde resin being such that a 50% by weight solution of the resin in water has a viscosity of at least 100 cP at 20°C, and coating the web with a melamine/formaldehyde condensation resin.

2. Process according to claim 1, wherein the resin mixture used for the impregnation is 50 to 90 weight percent urea/formaldehyde condensation resin, the balance being melamine/formaldehyde condensation resin.

3. Process according to claim 1 or 2, wherein the urea/formaldehyde molar ratio in the urea/formaldehyde resin in 1:1.5 to 1:2.5.

4. Process according to any one of the preceding claims wherein the melamine/formaldehyde molar ratio in the melamine/formaldehyde resin is 1:1.5 to 1:3.5.

5. Process according to any one of the preceding claims, wherein the web is a cellulose paper.

6. Process according to claim 1, substantially as hereinbefore described with reference to any one of Examples 1—3.

7. An impregnated and coated web obtained by a process according to any one of the preceding claims.

8. A method of decorative surface finishing of sheets of wooden material which comprises laminating a web according to claim 7, onto the surface of the sheet.

9. A method according to claim 8, substantially as herein before described with reference to Example 4 or 5.

10. A sheet of wooden material having a surface obtained by a process according to claim 8 or 9.

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